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QUARTERLY LETTER REPORT

COVERING

May 16, 1965 - August 15, 1965

THERMOCHEMISTRY OF SELECTED COMPOUNDS

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Submitted to:

Advanced Research Products Agency
The Pentagon, Room 3D-159
Washington 25, D.C.

Attn: Advanced Propellant Chemistry Office

September 10, 1965

MAJOR ACCOMPLISHMENTS

A. Aluminum Borohydride Hydrolysis

In this quarter we have continued to investigate the hydrolysis of aluminum borohydride with the vapors of water and hydrochloric acid, independently. In the latter case, (vapors of hydrochloric acid as the reactant) the generated heat data has become quite consistent. However, analytical determinations of boron and evolved hydrogen have continued to be low by an amount ranging from 10 to 20% of theoretical. In an attempt to verify our findings on these low values of boron, we sought the assistance of the Olin-Mathieson analytical laboratories in New Haven, Connecticut and their findings were in agreement with ours. An aluminum analysis was also made at Olin-Mathieson, but this was found to be greater than 100% of theoretical. This again is in line with our earlier findings, and it has since been found that the presence of hydrochloric acid in the bomb reacts to a slight degree with the bomb producing nickel ions which yield a high "aluminum" analysis. We have in this laboratory removed the interfering ions by a mercury cathode technique and the subsequent aluminum analyses have all been close to 100% of the theoretical amount. The mass spectrographic analysis of the evolved hydrogen was made at the Olin-Mathieson Company and a trace (0.1 mole percent) of pentaborane was found in the hydrogen. We have recently set up a more rigorous method of boron analysis. The apparatus is constructed of quartz and the method is that of the Chapin distillation. Although the procedure is more tedious, it is generally acknowledged as being more reliable.

Towards the end of this quarter we have begun to re-examine the products of the reaction of aluminum borohydride with water vapor. The boron analyses are beginning to appear to be much closer to the 100% of the theoretical value. The hydrogen, however, is still low but to a less extent. We will continue to emphasize the analytical support work with regard to this compound and its reaction with the vapors of water and hydrochloric acid vapors, respectively.

B. The Hydrolysis of SAP

Samples of SAP were prepared in a nitrogen drybox and these samples were allowed to react with water. Nine determinations of the heat of hydrolysis of the SAP material were made. The result gave a ΔH of -54.4 ± 1.4 kilocalories per mole. This uncertainty is equal to twice $\sim$. Analytical analysis of the resultant solution has shown the anion to be quantitatively intact. The cation, however, has been shown to break down during this reaction yielding a substantial amount of nitrogen gas. As yet, we do not have a quantitative number on this nitrogen but we are planning on getting that in the near future. The nitrogen determination was detected by the Olin-Mathieson laboratory by mass spectrographic methods. Other constituents of the cation have been quantitatively determined in this laboratory. We are seeking from Aerojet General a related compound the study of which may be helpful in gaining data with regard to the cation of the SAP compound.

PROBLEMS ENCOUNTERED

None.

ACTION REQUIRED BY ONR

None.

FUTURE PLANS

With regard to both the reactions of aluminum borohydride with the vapors of water and hydrochloric acid, respectively, and the reaction of the SAP compound with water, we plan on concentrating our efforts on the analytical phases of these reactions. Once having cleared up the mass balances of these reactions, we will return to recheck the related heat data from which we may then calculate the heats of formation of two compounds, SAP and aluminum borohydride.